

Sample Preparation: X-ray Powder Diffraction

1 Scope

This document provides the procedure for the sample preparation for x-ray powder diffraction (XRD) used by Geologist/Forensic Examiners within the Trace Evidence Unit (TEU). In x-ray powder diffraction, the material to be examined is typically reduced to a very fine powder and placed in a beam of monochromatic x-rays. Each particle of the powder is a tiny crystal or assemblage of smaller crystals, oriented at random with respect to the incident beam. The result is that every set of lattice planes will be capable of reflection. The material of interest must be have a regularly repeating atomic structure and should have been previously described using the Geologically-Derived Materials Examinations Procedures.

2 Equipment/Materials/Reagents

- Acetone, laboratory grade (or better)
- Agate mortar and pestle
- Chemical resistant gloves
- Deionized water
- Ethyl alcohol, laboratory grade (or better)
- Glass slides
- International Centre for Diffraction Data (ICDD) Powder Diffraction File (PDF)
- Laboratory coat
- Sieve set, to include 200 mesh sieve
- Additional materials may be used at the discretion of the Geologist/Forensic Examiner

3 Standards and Controls

Not applicable.

4 Sampling or Sample Selection

4.1 The collection and processing of materials with a regularly repeating atomic structure (typically crystalline materials) from submitted items of evidence can be reviewed in the TEU Evidence Processing Procedures.

4.2 At the discretion of the Geologist/Forensic Examiner, the entire sample or component(s) or mixes of components of interest may be analyzed. These materials are prepared

for identification as necessary at the discretion of the Geologist/Forensic Examiner. Individual sub-samples may not be representative of the entire specimen. Sub-samples are chosen based on the need to identify a particular component.

5 Procedure

5.1 Isolated Samples or Samples Which May Be Removed From Their Substrate

5.1.1 The sample or portion of a sample to be analyzed may be ground if necessary. To grind a sample or portion of a sample, transfer the portion to be analyzed to an agate mortar.

5.1.1.2 Grind the sample or portion of the sample with an agate pestle. Grinding method is determined as appropriate for sample at the discretion of the examiner.

5.1.1.2.1 Dry grinding – the sample is ground between mortar and pestle in air.

5.1.1.2.2 Wet grinding – the sample is ground between mortar and pestle in a carrying/lubricating medium. Medium should be easily volatilized, for example, acetone, water, or ethyl alcohol. Use enough medium to wet the sample and add more as necessary to keep the sample wet while grinding.

5.1.1.2.2.1 Take the sample to dryness when grinding is complete.

5.1.2 The sample or portion of a sample to be analyzed may be sieved if necessary. For most samples the portion of the sample which passes through a 200 mesh sieve is most appropriate for optimal XRD signal. Size fraction used is at the discretion of the Geologist/Forensic Examiner and is dependent on the individual sample.

5.1.3 Mount the sample in a holder. The holder used is determined at the discretion of the Geologist/Forensic Examiner as appropriate for the sample.

5.1.3.1 Low Background Slide

Evenly distribute the sample on a low background slide in the area illuminated by x-ray beam when the sample is analyzed.

5.1.3.1.1 Wet deposition.

5.1.3.1.1.1 Suspend the sample in a carrying medium. Medium should be easily volatilized, for example, acetone, water, or ethyl alcohol.

5.1.3.1.1.2 Place the suspension on a slide.

5.1.3.1.1.3 Evaporate the carrying medium.

5.1.3.1.2 Dry deposition. Sprinkle the sample onto a slide.

5.1.3.2 Well Holder

5.1.3.2.1 It is preferable to load the sample from the back side of the well holder.

5.1.3.2.1.1 Remove the back of the sample holder.

5.1.3.2.1.2 Place the holder front side down on a clean glass slide.

5.1.3.2.1.3 Pour the sample into the well and compress with a clean glass slide.

5.1.3.2.1.4 Replace the backing to the holder.

5.1.3.2.1.5 Invert the holder and remove the slide from the front of the holder, being careful not to disturb the surface of the sample.

5.1.3.2.2 To front load the well holder:

5.1.3.2.2.1 Pour the sample into the well holder.

5.1.3.2.2.2 Flatten the exposed side of the sample by gently compressing with a glass slide.

5.2 Samples Which May Not Be Separated From Their Substrate

5.2.1 Remove portion of sample and attached substrate or use entire sample with substrate for those materials that will fit on the sample holder.

5.2.2 Affix sample and substrate to sample holder.

6 Calculations

Not applicable.

7 Measurement Uncertainty

Not applicable.

8 Limitations

XRD analysis is limited to crystalline substances or materials with regularly repeating atomic structure.

9 Safety

9.1 Some carrying/lubricating media are classified as hazardous and will be handled and disposed of in accordance with manufacturer's recommendations and the FBI Laboratory Safety Manual.

9.2 Universal precautions will be used and at least the minimum appropriate personal protective equipment (PPE) such as laboratory coats and protective gloves will be worn when handling samples.

10 References

- Buhrke, Victor E., Ron Jenkins, and Deane K. Smith, *A practical Guide for the Preparation of Specimens for X-ray Fluorescence and X-ray Diffraction Analysis* (New York: Wiley-Vch, 1998).
- Cullity, B. D., *Elements of X-Ray Diffraction*, (Reading, Massachusetts: Addison-Wesley Publishing Company, Inc., 1978).
- Jenkins, Ron, and Robert L. Snyder, *Introduction to X-Ray Powder Diffractometry*, (New York, New York: John Wiley & Sons, Inc., 1996).
- Skoog, Douglas A., F. James Holler, and Timothy A. Nieman, *Principles of Instrumental Analysis, fifth edition* (Philadelphia, Pennsylvania: Harcourt Brace College Publishers. 1998).
- FBI Laboratory Safety Manual (current version).
- Geologically-Derived Materials Examinations, Trace Evidence Procedures Manual (current version)
- Trace Evidence Unit Evidence Processing Procedures, Trace Evidence Procedures Manual (current version)

Rev. #	Issue Date	History
1	02/07/18	<p>Updated title removing reference to TEU and throughout document where appropriate to reflect change from unit-specific to discipline-specific.</p> <p>Added Geologist/Forensic Examiner to Scope, Section 1 and throughout document where appropriate.</p> <p>Section 4 Calibration deleted, document renumbered.</p> <p>Section 4 updated heading to add "Sample Selection" and 7 retitled.</p> <p>Section 4.1 updated to reflect current title of document and update Section 4.2 to provide option for analysis of entire sample.</p> <p>Section 5 introductory sentence added and edits made throughout this section for clarity.</p> <p>Sections 9.1 and 9.2: 'will' substituted for 'should'.</p> <p>Section 9.2: 'Personal protective equipment' substituted for 'protective attire'.</p>
2	07/15/20	<p>References updated in Section 10 and throughout document.</p> <p>Capitalization of "X-ray" corrected throughout document.</p> <p>"Crystalline" deleted from Scope. "Typically" added to scope.</p> <p>Ether, chiller, and diffractometer deleted from Materials listing.</p> <p>"Or equivalent" changed to "or better" in Materials listing.</p> <p>Section 4.1, "crystalline" changed to "materials with a regularly repeating atomic structure." References cited were updated.</p> <p>Changed 'or' to 'and' in Section 9.1.</p> <p>Updated references throughout.</p>

Approval

Redacted - Signatures on File

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Date: 07/14/2020

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